	Puff	Char Line	°C
(Monitored)	8	40	860

Minutes of

SUPERVISORS' MEETING

April 26 - 27, 1956

Present: Dr. R. C. Sproull
Mr. A. E. O'Keeffe
Dr. C. V. Mace
Dr. R. B. Seligman
Mr. L. L. Long
Mr. J. T. Butler
Mr. J. Y. Mason, Jr.
Mr. S. W. Pleasants
Mr. J. C. Holmes
Mr. J. D. Hind
Mr. C. C. Cosby, Jr.
Mr. F. E. Resnik (Secretary)

ADMINISTRATIVE SESSION

Sheet Program

Dr. Mace reminded the supervisors that records of the percent of time, the budget expenditures and time in travel involved in connection with the sheet program should be kept. These records should be turned in once a week to Dr. Mace under project 2-006B. This will enable us to maintain records concerning the cost of our participation in the sheet program.

Purchase Requisitions

Mr. O'Keeffe reminded the supervisors of Dr. DuPuis' recent memorandum concerning the information which should be included on purchase requisitions. He pointed out that prices quoted on purchase requisitions should be firm discount terms and shipping information as concerns point of shipment and terms of freight billing should be included. It is also necessary that each requisition show the date desired for delivery of the item being requested. In connection with the delivery date, the following definitions were set up: when delivery date is specified as "ASAP", this means that delivery should be made as soon as possible consistent with normal channels of communication as, for instance, the use of letters for obtaining quotations and so on. Mr. O'Keeffe pointed out that follow up procedures on overdue items should be made through Mr. Price and that a detailed procedure will be worked out with Purchasing for handling this phase of the purchasing duties.

Studies of the menthol in Kools and Spuds from the comparison of brands showed that there was less variation in the menthol content of Spuds than Kools. Some of the Spud samples were up to 3 years old.

[redacted]

Vacations

Supervisors were reminded that vacation schedules should be set up prior to May 15. Each supervisor is responsible to insure that the vacation schedule leaves a sufficient number of people to maintain satisfactory working of his section at all times. It was pointed out that the normal vacation period is during the summer months. However, in the case of employees whose vacation should be tied with the factory vacation periods because of employment of husband or wife in the factory, each supervisor may schedule vacations at other times within the limitation above.

Visitors

Mr. Resnik pointed out that it is difficult to know exactly how to discuss the Research Department with visitors when their identity as concerns company connection is unknown. He requested that all visitors be introduced not only by name but by company affiliation and scientific background. In order to facilitate this situation, Dr. Sproull suggested that badges should be issued to all visitors in the laboratory which show the visitor's name, company affiliation and position.

TECHNICAL SESSION

PROJECTS:

1-001C Gas Chromatography (Gager, Murrill)

By chromatographing the smoke from five (5) cigarettes on a TCP-Celite column, those peaks that had previously appeared as minor constituents, now showed well defined curves at these higher concentrations. These are the later fractions peaking beyond 30 minutes and can now be investigated more thoroughly.

The first puff (after lighting) and the ninth puff from each of the cigarettes were collected in two different containers at liquid N_2 temperature. Both were chromatographed on TCP columns and their patterns recorded. Visually, they are different, but our experiences with variation in peak time with concentration of sample makes us withhold any conclusions until mass spectrometric analysis has been obtained from the various fractions. This may be an excellent way of concentrating trace quantities without contaminating the sample with large excesses of other constituents; that is, if qualitative differences do exist at different puff levels. This will be investigated fully.

The presence of many varied olefins, acetylenes, diolefins, etc. in the fractions identified by the mass spectrometer led us to check the effect of the hot tungsten filament in the

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section. No qualitative differences could be detected between the mass spectra from the two samples.

thermal conductivity cell. We must be sure that dehydrogenation is not taking place as the column's effluent gases pass that point. Hexane and 1-hexene have been chosen as representative samples. The results of the mass spectrometric analysis before and after passage thru the thermal conductivity cell show that dehydrogenation does not take place when these two compounds pass the hot tungsten filament. From these results it appears that the unsaturated compounds previously reported are from cigarette smoke.

Anticipating the sampling of smoke solids, to be run at elevated temperatures, a means for removing water from the samples was sought. Efforts were made to do this by non-chemical means and under as mild conditions as could be devised. It has been shown that a two inch section of Linde molecular sieve (#4A) placed on top of the chromatographic column within the heated zone will hold water and pass other materials of lesser polarity. This has been done with aqueous mixtures of suspensions of heptane, methyl n-propyl ketone, acetone and ethanol. Ordinary sample sizes of methanol do not pass the sieve under these conditions. Whether this is a function of the size of the sieve or of the polarity of methanol is unanswered at this time. The sieve did demonstrate that water can be withheld even at 100°C.

1-002D Cigarette Smoking Characteristics and Techniques (Hind)

Control cigarettes have now been smoked following the puff patterns of 15 subjects. Analysis of the material obtained has been for total tars and nicotine.

The results indicate that the relationship between smoke volume and total tars was linear, with the controls from 2/3 of the subjects being very close to an average linear plot.

Occasionally, very high results are reported for a tar determination, too high to be accounted for as "smoking error." We have decided to rerun all tar results which show deviations of more than 10% from an arithmetical average.

With improvements in apparatus, we have collected about 40 mg ± 4 mg from regular PM cigarettes smoked in 35 ml puffs with a one minute interval, the relationship between total volume of smoke and tars being consistent with the smoking results from 10 of the 15 human smoking patterns. The average value of 40 mg is higher than the result reported earlier which was 34 mg ± 3 mg for the standard smoke.

The results of the nicotine analyses have not been analyzed, principally because the results first obtained were seriously in error. This was due to the fact that the technician-analyst

Williams was undetectable with the infrared. It is felt that the samples analyzed were below the detectable limit for the infrared since considerable background interference was noted after the samples were concentrated.

did not understand the principle of the extraction procedure and eliminated an important step from the analysis. The results presently being obtained are believed to be much more reliable.

We have been continually improving the technique for collecting smoke from subjects. A new breathing tube is being fabricated, and arrangements have been made to permit raising or lowering the apparatus to fit the requirements of individuals. The spirometer tube (which fits over the cigarette) and the connecting air line is now automatically ventilated and little objectional odor is now associated with this part of the apparatus. In the next series of tests, the smoker will be relieved of the necessity of operating the foot switch.

UST 1-0020-3 Measurement of the Temperature of Burning Coal and Cigarette Smoke (Long)

A series of 25 tests were performed to measure the maximum temperature of the burning cigarette coal with thermocouples radially located through the cigarettes at Station 2. Another group of measurements were made at Station 6. In each case the smoking cycle was interrupted in order that the maximum temperature was obtained on a monitored puff.

The following results are mean values for each puff taken for 25 runs at Station 2:

	Puff Number	MM. Char Line	°C.
	1	0	123.3
(Monitored)	2	10.6	833.6

The moisture content for these cigarettes was 11.1%.

The following results are mean values for each puff taken for 25 runs at Station 6:

	Puff Number	MM. Char Line	°C.
	1	0	42.0
	2	5.4	62.8
	3	10.3	75.0
	4	15.2	85.0
	5	20.2	158.8
(Monitored)	6	30.6	828.2

The moisture content for these cigarettes was 11.2%

during the smoking procedure only three 35 ml puffs of smoke were collected in the evacuated flask. Usually an entire regular Philip Morris cigarette is smoked. Results of the latest analysis show little improvement over previous results.

A preliminary series of experiments were performed using the cigarette holder designed by the United States Testing Company, and the holder submitted by Philip Morris. The results indicated that a more practical holder was necessary in order to obtain a more normal manner of smoking for this study.

A holder has been designed and is being constructed for this investigation.

The results of the test indicate that the spirometer is operating satisfactorily.

1-002F Polyhydric Alcohols (Williams)

The wax obtained from the roaster flues in our plant was dissolved in a minimum of solvent (after purification) and chromatographed on filter paper. Ten microliters of this material failed to show the presence of TEG when run against a known. Fearing a second constituent was inhibiting the color development in the flue sample, known TEG and the flue sample were chromatographed as one spot. The known TEG became visible under these conditions. From this data, we conclude that the concentration of TEG present in the flue sample lies below the limits of detection by this method; if present at all. However, it must be pointed out that this was not the sample from which TEG was identified in the infrared. Infrared analysis of this sample also did not show the presence of TEG.

The method of quantitatively determining TEG by infrared analysis has shown variability with aliquots from a single solution. This is being studied by the instrument section. Until this is rectified no further samples will be submitted and all data obtained by the method must be held as suspect with this reservation; all values are minima.

An attempt was made to demonstrate the equilibration of TEG in a closed container. Ten grams of TEG on glass wool in a beaker was placed in a desiccator (no desiccant) along with a Petri dish containing dry glass wool and one containing glass wool moistened with 50 mls. of water. After 48 hours a material balance for TEG was sought. No TEG was found on the glass wool samples originally devoid of the humectant and no TEG was found in the washings from the desiccator. Ninety-seven percent of the TEG was recovered from the original sample vessel. This will be repeated in a desiccator at a constant moisture figure and with uncased tobacco replacing the dry glass wool.

Data are incomplete at present, therefore no conclusions can be drawn concerning the effect of the larger filter on the 20 port machine.

Dr. Sproull suggested that the glasswool be checked for the possibility of containing a lubricant on its surface. Dr. Mace suggested that in future studies of this type to use a ratio of 1 to 4 of TEG to H_2O since this is the ratio present in a cigarette.

TEG has been found to produce a large amount of derivative when treated with 2,4-dinitrophenylhydrazone. The derivative appears to be an osazone. Infrared and mass spectral analysis of the TEG sample show no indication of a carbonyl compound. This then presents the possibility of a new and novel means of analyzing for TEG. Mrs. Williams is investigating this possibility further.

1-002K Menthol (Carpenter)

Two approaches are being investigated for the determination of menthol in smoke: (1) the removal of color-contributing factors in non-mentholated smoke by the addition of bromine; (2) high temperature gas chromatography to remove methanol from the smoke solids. It has been shown that bromine does not affect the determination of known menthol. Its effect on non-mentholated smoke needs further investigation. This work is being withheld until the special mentholated cigarette determinations are completed. The results of this will be tabulated in a special report. One point should be mentioned, however; the reproducibility of the cigarette determinations was greatly improved. We attribute this to the pre-equilibration treatment given these production samples and recommend the practice for future menthol determinations especially where a taste preference is being codetermined.

1-003E Instrumental Research - Mass Spectrometer (Varsel, Resnik)

Considerable time has been spent during this period analyzing known compounds for the gas chromatography section so that retention times on these compounds might be established.

Mass spectra of some unique acetylene compounds (obtained from Experiment, Inc.) are being analyzed as reference patterns. Along this same line, examination of several gas chromatographic fractions did not show dehydrogenation after passing through a cell containing a tungsten filament. This means that the previously reported unsaturated compounds are coming from smoke and not being dehydrogenated by the filament. A more complete report of this experiment is covered under project 1-001C.

A sample of TEG was analyzed in the mass spectrometer to determine its purity. Introduction of 1 µl through the regular inlet system showed the sample to contain 0.65% H_2O and 0.35% other volatile materials. These trace amounts of

A paper chromatographic system has been established for a mixture of glucose, frutose and sucrose. Elution and concentration studies for quantitative determinations are being made. As yet no quantitative recoveries have been made. A loading of 200 micrograms can be run on Whatman #3 but this is the minimum quantity necessary for the infrared and high recovery must be made.

impurities could not be identified by this method of introduction. However, when 2 ml. of the TEG was carried through the non-volatile solvent technique the impurities could be detected easily. The impurities were tentatively identified as 2-methoxy-1-ethanol, 1,4-dioxolane, methanol, ethylene oxide, propylene oxide, methyl formate and carbon dioxide. This experiment points out the great advantage of using the non-volatile solvent technique for qualitative identifications.

Preliminary results show that the mass spectrometer is ideally suited for the analysis of aqueous solutions of glyoxal. The method is simple, accurate and rapid.

1-003E Instrumental Research - Electronics Group (Morrell, Harrow)

Considerable time has been spent during this period working with the thermistors for use in gas chromatography. High resistance thermistors (100,000 ohms) are being mounted in stainless steel cells and sealed with silicone rubber gaskets for high temperature gas chromatography. These high resistance thermistors are required because of the sharp decrease in resistance with an increase in temperature. The reference cell will be mounted at the input of the column and will be packed with glass beads to provide more intimate contact with the gas to increase the heating efficiency.

Attempts to crimp the 0.001" platinum leads from the high resistance thermistors into a piece of 0.006" stainless steel capillary tubing resulted in the breaking of the leads. This technique has been replaced by silver soldering which works very satisfactorily.

Sensitivity tests in the gas chromatography column of these high resistance thermistors showed them to be far superior to the low resistance thermistors at high temperatures.

1-003F Instrumental Research - Absorption Spectroscopy (Harrow, Resnik)

1. Lyophilization Technique

The lyophilization technique for obtaining infrared spectra of amino acids, sugars, starches, etc., is being continued.

Along this same line, attempts are being made to lyophilize non-polar materials quantitatively. The problem is to obtain a homogenous mixture of a non-polar compound such as anthracene and a polar material such as KBr.

Curing Study

A comparison of tobaccos cured by a coal stoker, steam heated air and oil systems has been completed. The coal stoker system produced a tobacco having a significantly

[redacted]

Preliminary attempts were made to lyophilize anthracene from a benzene solution using ethanol, dioxane, or water to dissolve the KBr. The best results were obtained when the KBr was added to the benzene solution of anthracene followed by lyophilization. Water was then added to the sample and it was again lyophilized. The infrared spectrum of this disk showed a good transmission. An attempt will be made to make this technique of analyzing a non-polar material quantitative.

2. Total Carbonyl

In an attempt to develop a method for measuring total carbonyl compounds in cigarette smoke an investigation of the basic curves of the 2,4-DNP derivatives was carried out.

The basic curve of 2,4-DNPH reagent showed an extremely low absorbance. The area under the curve from 360 to 600 mu was calculated and plotted vs. concentration.

This same plot was carried out on the basic curves of the 2,4-DNPs of acetaldehyde, propionaldehyde, diethyl ketone, acetone and furfural. A plot of the area vs. concentration on all these carbonyls followed Beer's Law. All the compounds fell on the same Beer's Law plot with the exception of furfural.

The literature reports that these four compounds make up 86% of the carbonyls in cigarette smoke while furfural only accounts for 3% of the carbonyl content. Methyl ethyl ketone and formaldehyde make up the remainder being present as 10% and 1% respectively.

Therefore, if upon the analysis of the 2,4-DNP of methyl ethyl ketone the curve falls on this established Beer's Law plot, a method for measuring total carbonyl in cigarette smoke may be possible. This will mean that at least 96% of the monocarbonyl compounds will fall on the same Beer's Law plot.

In an attempt to determine the effect of neglecting the excess reagent a mixture of a known amount of the 2,4-DNP of acetaldehyde and 2,4-DNPH reagent was analyzed. The sample contained 12 µg/ml of acetaldehyde plus 12 µg/ml of reagent. The area under the curve was calculated, the concentration read from the graph was found to be 12.7 µg acetaldehyde per ml. A second sample containing 12 µg/ml of acetaldehyde and 8 µg/ml reagent gave a result of 13.2 µg of acetaldehyde per ml. These results show that the carbonyl found is a little high if the excess reagent is disregarded. It was suggested by Dr. Spreull that this reagent could be compensated for from its Beer's Law plot.

Dust Loss

As reported previously, a method for measuring the loose dust on the sheet surface is under development. A device for brushing the sheet uniformly is being designed.

Plans are to analyze the 2,4-DNP of methyl ethyl ketone. A mixture of 5 or 6 2,4-DNP derivatives with excess reagent will be analyzed to determine the reproducibility of the method.

3. Miscellaneous

TEG

The TEG determination by infrared has shown variability with aliquots from the same TEG standard. It is possible that this may be due to allowing the samples to remain on the lyophilization apparatus after they have reached room temperature. This non-reproducibility will be further investigated.

The dye on the Marlboro pack has been tentatively identified as a rhodamine or fluorescein dye by the U.V. Lack of I.R. reference patterns of dyes has made further identification impossible.

Work on the polyphenols in tobacco is being continued with emphasis on chlorogenic acid.

I.R. spectra can be obtained from the micro KBr die but further work is required before micro quantities can be analyzed.

1-003J Infrared and Ultraviolet Studies of Smoke (Harrow, Resnik)

Statistical analysis of the CO/CO_2 data from cigarette smoke from five different brands showed day to day effects that could not be explained. Mr. Budne said no definite conclusions could be drawn and recommended an experiment to eliminate these day to day effects.

Work is now in progress to smoke one cigarette from each of five brands: Lucky Strike, Chesterfield, Camel, Old Gold and Philip Morris. A second set of the 5 brands will be smoked starting with Chesterfield and this rotation continued until 40 cigarettes are smoked. This work will require about 3 weeks to complete.

1-003K Smoke Comparison with the Mass Spectrometer (Varsel, Resnik)

With the installation of the micromanometer, studies are again underway to develop a method for measuring moisture in cigarette smoke.

The smoke is being collected by metering a 35 ml. volume between two large-bore stopcocks followed by introduction into a 1 liter evacuated flask. Considerable difficulty has

been encountered because of the condensation of the tars and moisture during smoking, especially between the metering stopcocks. Analysis of the bulb plus the volume between the two stopcocks showed a gradually decreasing moisture content during a series of analyses from the same sample. Results varied from 15 mg to 2 mg H_2O per cigarette. Analysis of the bulb alone was reproducible (approximately 2 mg/cigarette) within a sample and even from cigarette to cigarette.

These results showed that moisture can be determined in smoke in the mass spectrometer if a homogenous sample can be obtained.

In an attempt to eliminate this moisture condensation and obtain a homogenous sample the space between the two stopcocks was heated during smoking. Results showed that this did not eliminate the variations in moisture content.

Perhaps a higher temperature is required.

Several suggestions were made to eliminate this condensation. One suggestion was to collect only three puffs of smoke; however, it was pointed out that it would be difficult to decide which puff to collect and to convert the moisture back to a per cigarette basis. Another suggestion was to increase the collection volume from 1 liter to 3 liters. It was also pointed out that it might be possible to drive all the tars into the bulb by heating the "metering volume" and cooling the collection bulb after a cigarette has been smoked. The sample could then be cooled in liquid nitrogen, the air pumped off and the sample analyzed after it had reached room temperature.

1-005B Evaluation of Filter Materials (Long, Scherr)

Three samples of Celanese tow have been received: (1) 75,000 total denier, 5 dpf; a duplicate of our present Marlboro tow; (2) 80,000 total denier, 3 dpf and (3) 90,000 total denier, 3 dpf. The latter two samples were designed for 25 - 30% F.E. in 10 mm plugs (for LSP cigarette). These two samples were made into 60 mm plugs 4-26-56. The evaluation of the samples in LSP cigarettes should be complete shortly.

1-005C Physical Testing Methods and Results (Long, Scherr)

The large diameter total filters are ready for evaluation on the 20 port smoking machine as soon as it is available.

No other work is being done on this project pending completion of the comparison of brands smoking.

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2-001 Aging of Tobacco (Mason, Crayton)

Natural

The April sampling of the five hogsheads of 1955 tobacco has been completed. The present samples appeared to be drier and more brittle than in December 1955 though the moisture contents were similar (10.06 and 10.92%). The chemical analyses of these samples are in progress and the results will be compared to those obtained in December.

The IR and UV spectra of the oil, resin and wax fractions of the December, initial, samples of 1955 tobacco are similar to those of the 1954 crop, first sampling.

Forced

Boxes packed at normal, 25% greater than normal hogshead density and normal density wrapped in aluminum foil are being forced aged. These boxes will undergo six sweating periods of ten days each at 95°F and 70% R.H. The larger number of sweating periods should produce a better aged tobacco. The shorter sweating period should not materially effect the aging process.

The tobaccos previously forced aged showed the following differences after three sweats. There was a decrease in the ash and nicotine contents of all samples. The tobaccos packed at normal and 25% greater than normal density in regular boxes showed a decrease in nitrogen and that packed in perforated and wrapped boxes an increase in sugar. The smoking characteristics of these tobaccos are being determined.

Tobacco samples similar to those discussed above were extracted with petroleum ether. The petroleum ether extract was sprayed on glass wool, the ether allowed to evaporate, and the extract forced aged. When compared to the unaged extract the differences in aroma were fairly similar to those found in tobacco.

Both the aged and unaged extract is being extracted from the glass wool and the oils, resins and waxes will be compared.

In the discussion which followed these results on the aging studies, Dr. Seligman suggested that the forced aging be investigated by aging some tobacco in a CO_2 atmosphere. Dr. Mace recommended a photographic record of the tobacco in its various sweating stages. It was also pointed out that the resins and polyphenols need investigation of the determination of quality in tobacco.

2-001B Chromatographic Aging Studies (Gager, Edmonds, Greene)

Paper chromatographic systems have been established for amino acids and alkaloids. The systems for sugars are becoming satisfactory and a sensitive spray reagent has been developed. The sugars separate on ascending development but increased resolution is desired before quantitative analysis is attempted. For that reason, descending development is being examined. Also sample loading will be investigated to try to reach the concentration level required for quantitative infrared analysis. The effect of elution from the paper strip will be studied from the standpoint of cellulose contribution and percent recovery.

One fact that became evident from the chromatography of the known sugars is that the samples are not pure. Most of the monosaccharides are contaminated with what appear to be disaccharides. One sample of glucose (NBS) was chromatographically pure. Thus, the standard IR curves may contain contaminants if the concentration of impurity is in its level of detection. Chromatographically pure samples should be obtained for standards either by purchase or other means. Dr. Sproull pointed out that considerable work has been carried out at Ohio State on the chemistry of sugars.

Varying temperature may be a factor to be reckoned with in all our paper strip chromatography. This may require enclosing the chambers for the accuracy and reproducibility needed for quantitative work.

2-003B Continuous Moisture Meter, University of Virginia (Long, Super)

As mentioned previously, experiments using the mainstream of tobacco were tried. These experiments were unsuccessful since we were not able to get the tobacco to flow through the cell. The instrument has been removed from the factory.

Work on this project will be discontinued until further notice.

2-003C Equilibrium Moisture Studies (Mason, Sharp)

Correlation coefficients between the equilibrium moisture contents and the chemical composition of the second set, TRP3, of burley samples at 77°F and 40% R.H. have been determined. The relationships are similar to those reported for the TRP1 samples

At 70% R.H. the soluble constituents, nitrogenous materials and water soluble acids have a positive effect on equilibrium moisture content while the cell wall and inorganic constituents have a negative effect. At 40% R.H., the relationships are reversed. This could in part explain the abnormal behavior of burley tobacco at 20-40% R.H.

2-005 Evaluation of Tobacco (Mason, Sharp)

N. C. State Evaluation Test

The statistical analysis of the ash results shows that Coker 139 and 2041-4 had larger ash contents than the DB 101 control and DB 244 which were similar. The statistical analysis of the results of the petroleum ether extract contents is in progress.

Official Variety Test (Rural Hall Experiment Station)

The following chemical differences between White Gold, Yellow Special A, Hicks, Va. 21, Golden Cure and DB 101 have been observed. Varieties White Gold and Yellow Special A had a smaller nicotine content than the DB 101 control. All varieties had a smaller sugar, ash and nitrogen content than DB 101 and Hicks a larger petroleum ether extract content than the control.

Va. 21, Hicks and Golden Cure appeared to be superior in overall chemical composition. The smoking characteristics of these varieties were also believed to be similar and more satisfactory than the other varieties.

Sucker Control Test

No significant differences in chemical composition were observed between the tobaccos not topped or suckered, topped and suckered by hand, oil and maleic hydrazide. The failure to observe smaller nicotine, nitrogen, T.V.B., and ash contents normally associated with no topping and suckering could be due to a sampling error. The L.S.D. values for all determinations were larger than normal.

2-006B Tobacco Sheet (Binder Studies) (Hind)

Materials are being collected for the intensive study of various binders proposed for use in the sheet program. Various methods for the assay of technical glyoxal solutions are being invested.

2-006B Tobacco Sheet (Long, Super)

American Machine & Foundry Pilot Plant Studies

As mentioned at the last meeting preparation of a control sheet was being delayed due to operational difficulties. Most of the difficulties were centered around the overdust feeder.

During the past two weeks, temperary measures were taken which overcame most of the problems with this feeder. Meanwhile a new feeder is under design by the AMF Engineering Department.

After completion of the alterations on the overdust feeder the plant was operated for three days without serious difficulty. The sheet made during this period looked quite good, however, the binder content was approximately 12% which is on the high side of the allowed range. Fifty pounds of sheet from this production is being sent to us as a control. The operational and sheet data are being sent with the sheet.

Samples using standard binder with increased glycerine content, medium viscosity CMC, low viscosity binder - water added, flax pulp, etc. are under preparation.

During the course of preparing for runs on these samples, it was observed that the viscosity of the binder was quite variable, particularly from one batch to the next. Factors affecting the viscosity of the binder should be determined since this factor is important in the spraying operation.

These samples are being made as part of our studies on the improvement of the physical properties of sheet.

Additional studies incorporating flavors in the sheet, tobacco stems as pulp in the binder and processed stems as the binder are planned.

Film Coating (by means of spraying)

Methods of forming films of the MTS binder are under development for use in laboratory studies on sheet binders. To date, we have been able to form satisfactory films with the regular MTS binder and with a diluted binder. Film from the binder in the range of 1 gm/ft.^{2} has exhibited a tensile strength of about 0.9 kg/in.

Efforts to form films with guar gum and gelatinized stems individually have not been successful to date due to the fact that they become gummy when remoistened for removal from the coating plates. Methods of removing the films from the plates will be studied.

A film composed of a combination of guar gum and gelatinized stems was satisfactorily formed. This film had a weight of 0.98 gms/ft.^{2} and a tensile strength of 0.55 kg/in.

Dust Loss

Experiments were repeated in the utilization of honey as a means of reducing dust loss and breakage. These new experiments were comparable to the previous results; i.e., 2 - 8% pick-up produces reduction in dust loss of about 70% and a reduction in breakage of about 50%.

These experiments will be discontinued unless the pilot plant studies indicate a need for further work along these lines.

An attempt is being made to correlate sheet "dustiness" with dust loss. Such a correlation will require a method for measuring the "dustiness" of the sheet (amount of loose dust on the surface of the sheet.)

Preliminary experiments using a hand brush for removing the loose dust indicates that this dust contributes considerably to the dust loss as now measured. This also indicates that loose dust formed as a result of cutting. An apparatus using a rotary brush which will be arranged to apply a uniform pressure is under construction for use in measuring "dustiness".

Compressed Sheet

This work is being delayed pending the construction of a new die.

Miscellaneous

Precise picking of L & M cigarettes shows a sheet content of about 5%.

2-006C Physical Evaluation of Tobacco Materials (Long, Super)

1) A standard method for the particle size analysis of sheet raw materials is under development. This method should be ready shortly.

2) Measurement of the particle size of dust ground for pilot plant studies (discussed above) shows that most of the material is less than 100 mesh although 50% of the material is ground to pass through 50 mesh and the other half through 80 mesh.

2-007F Specifications and Service to Production (Long, Scherr)

Nothing new to report during this period.

2-014 Miscellaneous Development Investigations (Long, Scherr)

Regular PM cigarettes were conditioned at 75°F and 60% R.H. then selected in the weight range of 103-105 cigarette/4oz. These cigarettes were then allowed to condition at various humidities for several days.

The results were as follows:

R.H.	45 %	56 %	66 %	71 %	75 %	81 %
Moisture	9.8	11.1	14.0	16.0	17.5	19.5

Ten cigarettes from each group were securely packed and sealed in glass jars for shipment to the Battelle Memorial Institute. They have an apparatus which may be applicable for moisture sensing.

4-002 Comparison of Brands (Sowell, Cosby)

The physical and chemical tests on the comparison of brands cigarettes picked up in Harrisburg, Pennsylvania is progressing satisfactorily. Moisture, individual weights and package inspection tests have been completed.

4-003A Leaf Analysis (Pleasants, Wiley)

A. The following analyses were completed on leaf and filler:

125	Nicotine
270	Petroleum ether
222	Ash
72	Total Nitrogen
480	T.V.B.
6	Sands
103	Sugar
376	Moisture

4-003D Miscellaneous Laboratory Tests (Butler, Clarke)

Since our existing methods for the analytical determination of the nitrogen containing compounds in tobacco do not cover all its nitrogenous constituents, a scheme of

analyses is being adopted for their classification. This scheme involves a stepwise procedure for the removal of definite nitrogenous fractions and the determination of the compounds in these fractions.

4-003D Moisture Determination in Tobacco

A new method of applying known amounts of water to the heated columns has been tried. Preliminary tests show a possibility of giving increasing areas under the curves with increasing amounts of water added to the column. The areas under the curves are being checked by using a planimeter. Additional tests will be necessary before definitely knowing if there is a linear relationship.

Determination of Tars in smoke

Several samples of smoke in ethanol toluene were read on the Klett in an attempt to determine tars in smoke colorimetrically. The results indicated that there was very little difference in Klett units between samples of high and low tar content and it would not be feasible to use a colorimetric method.

Dr. Seligman suggested that tars might be determined using a fluorescence spectrophotometer.

Marlboro Box

In order to assist the Sales Department, a problem on "color rub-off" from the Marlboro box is under study.

Red ink on the Marlboro box was dissolved in chloroform and run on the Cary. It was tentatively identified as a rhodamine or fluoresoein dye.

Boxes made by United, Downing, Zumhill and Marathon were examined for whiteness of paper and for brightness and coverage of red ink. The Marathon and United boxes were definitely the whitest, and Downing and Zumhill were both slightly yellow. United used the brightest ink and United and Marathon had the most even coverage. No definite statement as to the difference in fluff off can be made because the boxes had been handled too much before we received them.

Polyphenols

The materials precipitated by lead in the clarification of tobacco extract in the sugar determination are being examined in order to get some idea of the qualitative composition of polyphenols.

4-004A Smoking Machines (Sowell, Cosby)

A. Both our 8 port and 20 port smoking machines were operated during this period and the following analyses were completed.

20 port	152	Tar analyses
	144	Furfural in smoke
	128	Nicotine in Smoke
8 port	8	Tar analyses

B. The following analyses on smoke solutions for Comparison of Brands were completed:

152	Tars
128	Nicotine in smoke
144	Furfural in smoke

Special analyses for Jack Hind

41	Tars
23	Nicotine

Special analyses for Bob Carpenter smoked on 8 port smoking machine

8	Samples

cc: Dr. R. N. DuPuis
Dr. S. M. Cantor